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INTERIM SCIENTIFIC REPORT

ADVANCED TECHNOLOGY OF INTERCONNECTIONS IN MICROELECTRONICS

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## SECTION I

### 1.0 INTRODUCTION

The performance objectives of this program are to conduct the following investigations:

- Item 1 - Investigate advanced interconnection techniques for silicon monolithic circuits from a metallurgical viewpoint to improve reliability. This could include the following:
  - (a) A critical analysis of metal diffusion into active areas of the device.
  - (b) Adhesion properties.
  - (c) Diffusion from substrate into metal films.
  - (d) Degradation of film from bonded leads.
  - (e) Defects in substrate (scratches, uneven oxide).
  - (f) Film thickness requirements.
  - (g) Characterization of various metal systems from standpoint of optimum device characteristics and reliability.
- Item 2 - Characterize and identify interconnection failures.
- Item 3 - Conduct a thorough investigation of the nature of microcircuit bonds produced by soldering, welding ultrasonic bonding and diffusion bonding processes. This investigation should include the metallurgical, physical and analytical aspects of bonds produced by various bonding techniques to determine the structure of bonds, interface situations, etc.

From this study optimum bonding techniques will be established to enable industry to consistently obtain the type of interconnection bond required by NASA, and at a cost acceptable to both industry and NASA.

## SECTION II

### 2.0 SUMMARY OF EFFORT

- (1) Standard vacuum evaporation procedures and low energy inert gas sputtering procedures have been developed and evaluated for the aluminum, molybdenum-gold, and chromium-gold metallization systems.
- (2) The residual gas ambient of the high vacuum evaporation has been determined.
- (3) High current density tests have been conducted with the aluminum, chromium-gold, and molybdenum-gold metallization systems.
- (4) The limitations of the chromium-gold metallization system have been investigated and as the result, the chromium-gold system is not recommended for use in high reliability integrated circuit technology requiring high temperature processing.
- (5) The molybdenum-gold metallization system has been investigated. The results indicate that the molybdenum-gold system is superior to any other metallization system evaluated to date.

## SECTION III

### 3.0 VACUUM EVAPORATION PROCEDURES

Standard vacuum evaporation cycles were established for aluminum, chromium-gold, and molybdenum-gold metallizations. The parameters which were standardized included the substrate heating cycle, substrate temperatures, deposition rate, filament positioning, and substrate cooling schedule. Since increased deposition times have been used in some experiments, an attempt was made to measure the radiation heating of the wafer. Using surface thermometers, a simulated evaporation run gave a measured 140°C rise in 5 minutes. During the "quick cool," the substrate block temperature (as indicated by the internal thermocouple) fell more rapidly than the surface or wafer temperature. Modifications were made in several of the vacuum evaporators such that the temperatures measured were more representative of the true wafer temperatures. MOS devices and sensitive matched beta transistor pairs were used to develop and evaluate the standard deposition procedures.

From these experiments, the following conclusions are drawn:

- (1) The MOS device and the matched beta transistors require similar deposition conditions to make "good" devices. Both are sensitive to deposition conditions.
- (2) The indicated thermocouple temperatures are considerably higher than the actual surface temperatures in the normal evaporator, when no radiation source is present.
- (3) The time the wafer is exposed to the radiation from the evaporating source, the source size, temperature, and distance from the substrate are critical variables.

Surface temperatures far in excess of those indicated by the thermocouple are possible during the deposition.

- (4) The "quick cooling" cycles are generally ineffective in rapidly lowering the wafer temperatures. If "quick cooling" is desired, an inert gas backfill is necessary.
- (5) The predeposition substrate temperature is a less critical factor, as long as some minimum temperature to adequately "clean" the surface is reached. This minimum value is between 100° and 200°C in these experiments. Depositions at or below 100°C have generally shown poor adherence.
- (6) Using controlled conditions determined by experiments, it is possible to produce "good" devices in any normal vacuum deposition system. It is necessary to closely control the radiation heating of the wafer, particularly during the first "hot" deposition.

## SECTION IV

### 4.0 RESIDUAL GAS ANALYSES OF THE HIGH VACUUM EVAPORATION SYSTEM

A part of the instrumentation on the vacuum evaporator is a Residual Gas Analyzer. This is normally used for routine leak checking of the system, but periodically, a complete analysis of the residual gas is performed. Typical analyses results are shown in Table I.

TABLE I  
RESIDUAL GAS ANALYSES OF NRC EVAPORATOR

	Total Pressure	H <sub>2</sub> O	CO	N <sub>2</sub>	CO <sub>2</sub>	O <sub>2</sub>
8-10-66	4x10 <sup>-7</sup> T	76%		15.3	7.0	1.6
11-28-66	3x10 <sup>-7</sup> T	70		17	9.5	3.5
11-28-66	1x10 <sup>-7</sup> T	60		26	9.2	4.8
1-3-67	1x10 <sup>-7</sup> T	33.5	52	11.1	1.8	1.6

These analyses are with the substrate and filaments all at room temperature. (The nitrogen and carbon monoxide components are determined from the mass 28 peak, and are combined in the earlier analysis.) On heating of any portion of the system, an increased pressure is seen, which is primarily water vapor.

No gas analyses are available for the evaporator currently being used for the chromium-gold depositions. This is an ion pumped system with a cryogenic titanium sublimation pump. Although it uses an unbaked glass bell jar, pressures are less than 10<sup>-7</sup> torr range during the depositions. Water vapor is probably the main component of the residual gas in this system.

## SECTION V

### 5.0 THE ALUMINUM METALLIZATION SYSTEM

Aluminum is the material that is most commonly used as an interconnection metal for silicon transistors and integrated circuits. It is easily applied by high vacuum evaporation techniques and adheres well to the  $\text{SiO}_2$  surface of metal overlay devices and integrated circuits, in addition to affording good ohmic contact with both P and degenerate N type silicon. It is easily patterned, and contacted with gold or aluminum wires. Aluminum does, however, have certain limitations, some of which are listed below:

- (1) At elevated temperatures, aluminum and gold interact to form various intermetallic compounds which increase contact resistance and can result in open bonds.
- (2) When a high density current (above about  $10^6 \text{ A/cm}^2$ ) is passed through an aluminum interconnection stripe, mass migration of the aluminum can be seen toward the positive terminal.
- (3) Above  $500^\circ\text{C}$ , the aluminum attacks the underlying  $\text{SiO}_2$  rapidly.
- (4) Although satisfactory MOS devices can be made with aluminum metal, it is virtually impossible to obtain, in combination, all of the most desirable MOS properties.
- (5) Although aluminum is inherently about as conductive as gold, it is very sensitive to oxygen or water vapor in the vacuum system during deposition and so, generally, has a higher sheet resistance.



Although aluminum metal is not of primary concern for this contract, all subsequent metallization systems must be related back to an aluminum system in order that a common denominator be available for the evaluations. Hence, some of the properties of a particular type of aluminum metallization will be discussed from time to time in reports on this contract, but always in the context of comparison with a particular advanced metallization system.

#### 5.1 HIGH CURRENT DENSITY TESTS WITH ALUMINUM METALLIZATIONS

Aluminum metallization has been studied as a reference system and specific data is available on the high current density tests. These results are summarized in Table II.

TABLE II  
MEAN TIME TO FAILURE FOR ALUMINUM METALLIZATION AS A  
FUNCTION OF RESISTIVITY AND CURRENT DENSITY  
(AMBIENT = 175°C, 50% CONFIDENCE LEVEL)

J = (10 <sup>6</sup> A/cm <sup>2</sup> )	0.5	1.0	1.2	1.5	2.0	2.8
$\rho = 2.6 \mu\Omega\text{-cm}^*$	---	1500hrs	---	---	69	---
$\rho = 4.0 \mu\Omega\text{-cm}$	---	470	---	---	25	---
$\rho = 6.0 \mu\Omega\text{-cm}$	---	130	---	---	13	---
All available data	480	560	17	20	30	4

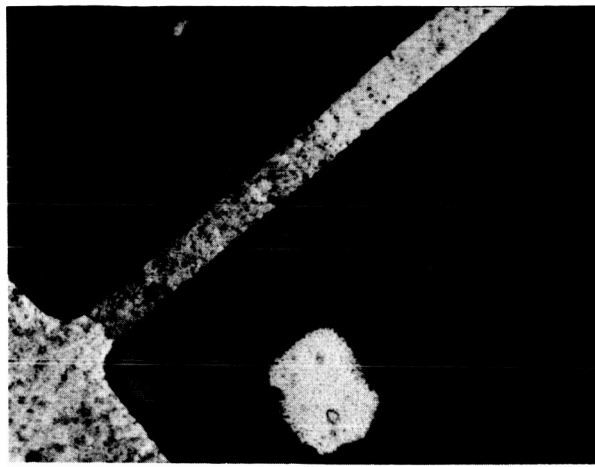
\*Resistivity varied by deposition rate.

Examination of the failures show a single dominating failure mode, i.e., a random opening of the interconnection, occurring in a porous rough-textured metal. During the processing, the aluminum is subjected to 450°C for perhaps 30 minutes, and changes from a smooth, nonporous film, beginning to develop a characteristic roughness. This roughness and porosity can be increased by subsequent thermal treatments. The intentional addition of moisture to the dry air furnace

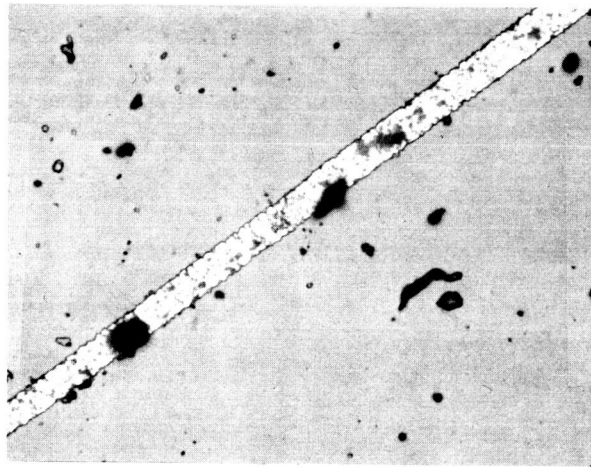
had no significant effect on unmounted samples. The same rough texture appears in the high current density tests in dry N<sub>2</sub> sealed TO-5 cans, although to a greater degree. The proposed failure mechanism is that a thermal reaction causes a porosity and reduced cross-sectional area, with a localized increased current density. This causes localized hot spots and accelerated reaction at these sites. Failure occurs when the voids form a continuous break across the strip. This is illustrated in Figure 1. The ½-mil strip at the top was heated 48 hours at 450°C in wet air. The middle sample was a failure occurring after 1150 hours at 10<sup>6</sup> A/cm<sup>2</sup> with an oven temperature of 175°C. The 1-mil line, adjacent to the failed ½-mil strip, carried no current during the test and is shown at the bottom of the figure.

Additional experiments have been performed to demonstrate the thermal nature of the reaction. A sample of 10 units (using the ½-mil lines) was tested at  $2 \times 10^6$  A/cm<sup>2</sup> and an oven temperature of 175°C. These units had normal processing, i.e., gold backing, bonding, and canning in TO-5 cans. The mean time to failure was 25 hours. A second group of 10 units was prebaked at 450°C for 2 hours prior to the high current tests. These units has a mean time to failure of 24 hours. A third group of 10 units was prebaked 8 hours at 450°C before the high current tests. The mean time to failure was reduced to 15 hours.

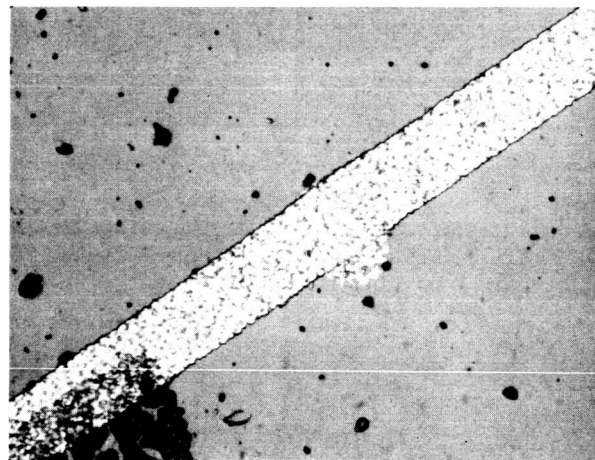
This brief study of aluminum metallization has included vacuum evaporated metal using tungsten filaments, tantalum filaments, and electron beam heating. Both normal (10<sup>-6</sup> torr) vacuum and high (10<sup>-8</sup> torr) vacuum samples have been included. The films are approximately 6000 Å thick, and are generally deposited in from 1 to 10 minutes. All the aluminum tested showed the thermal changes described. Other workers have reported different mechanisms, such as strip cracking (which appears to be a grain boundary phenomena)



800X  
0.5-Mil Line After 48 hrs 450°C



800X 0.5-Mil Line  $10^6$  A/cm<sup>2</sup> 1150 hrs  
Film Temperature ~180°C



800X 1-Mil Line - No Current

Figure 1. Photomicrographs of Aluminum Lines

and migration of the metal from the negative end toward the positive terminal. Neither of these have been observed so far in this program.

## SECTION VI

### 6.0 REQUIREMENTS OF A MULTILAYER METALLIZATION SYSTEM

To overcome some (or all) of the limitations imposed on the interconnection system by the use of aluminum, advanced multilayer systems are being investigated as the basis for this program. Most of them are two layer systems, although several more complex "sandwich" structures have shown promise.

Whatever the structure or complexity of the multilayer metallization system, it must fulfill many basic requirements if it is to find successful application. These are listed below:

- (1) The multilayer metallization system must permit patterning of geometries, by photoresist techniques, down to the smallest dimensions necessary for current integrated circuit practice.
- (2) Not only must the base film of a multilayer metallization pattern adhere well to the oxide, but each succeeding layer must adhere well to the one below it.
- (3) The electrical conductivity of any new metallization system should, for the same film thickness, be as high as aluminum.
- (4) Whatever metal makes contact to the silicon, it must have the capability of making a very low resistance ohmic contact.

- (5) It is possible for two adjacent metals in a multilayer metallization system to react at high temperature and form intermetallic compounds that will reduce adhesive strength or conductivity, or rupture film.
- (6) Since aluminum appears to have a fundamental limitation with respect to material migration at current densities, we look to these advanced metallization systems to provide interconnections for the high current density applications.

## SECTION VII

### 7.0 MULTILAYER SYSTEMS FOR INTEGRATED CIRCUIT INTERCONNECTION

In a survey of combinations of reactive metals with an overlay of another metal, a pattern consisting of a narrow strip ( $\frac{1}{2}$  mil wide and 54 mils long) was used. The strip was terminated in large bonding pads of the same metallization. From the measured thickness (generally 3000 to 6000 Å), the resistivity for each combination was calculated from the measured resistance. The theoretical resistivities for the metals vary from 1.6 microhm-cm for copper to 4.8 microhm-cm for molybdenum. The multilayer strips should then be between these values. However, it is often found that theoretical resistivity is not obtained in the normal vacuum deposited films. In these experiments, values of about twice the theoretical resistivities were generally the best obtainable for the single metals. In some cases, the multilayer films showed a satisfactory resistivity as deposited, but upon heat treating in subsequent processing the resistance rose rapidly, evidence of an intermetallic compound forming. This was observed, for example, with nickel-aluminum films. In other cases, the intermetallic was formed during the deposition at 260°C, as with titanium-nickel films.

Table III shows a range of bulk resistivities measured for a number of metal combinations that have been investigated. Although copper looks promising from a conductivity standpoint, in thin film form it oxidized badly upon exposure to even moderate temperatures for a short time.

From the results obtained so far, two systems have been selected for intensive study. They are the chromium-gold and molybdenum-gold structures.

TABLE III  
RESISTIVITY IN MICROHM-CM FOR SINGLE AND MULTILAYER FILMS

First Layer Second Layer	Aluminum	Chromium	Molybdenum	Titanium	Nickel
Aluminum	2.8 to 5.8	3.0	39	3.4*	7.7
Nickel	See Ni-Al	125 to 129	16.9 to 20.0	112 to 121	7.8
Copper	-	5.7	3.3 to 6.7	4.0 to 6.0	---
Copper-Gold	-	2.9 to 3.4	5.6	12	12
Gold	-	2.9 to 15	2.7 to 7.0	4.6, 4.8, 5.2	16.8
Titanium	See Ti-Al	-	-	90	See Ti-Ni

\*Thickness measured only approximately.



## SECTION VIII

### 8.0 CHROMIUM-GOLD

Chromium-gold metallization is a promising substitute for aluminum as used in the interconnection metallization of transistors and integrated circuits. Chromium adheres well to glass layers and will provide ohmic contacts to both P and N type silicon if the surface regions are degenerately doped by appropriate diffusions. In this and the other multimetal systems studied, the resistivity of the metallization is studied by preparing a pattern consisting of a narrow strip ( $\frac{1}{2}$  mil wide and 54 mils long). The strip is terminated in large bonding pads of the same metallization. From the measured thickness (generally 3000 to 6000 Å) the bulk resistivity is calculated from the average measured resistance. Ohmic contact to silicon is checked by depositing the metallization onto silicon wafers with 4 x 5 mil openings etched through a protective oxide 10,000 Å thick. The resistance between adjoining metallized contacts is measured with a sensitive bridge. The sheet resistance and nature of the wire bond to the metallization is studied by measuring the resistance between two leads attached to opposite sides of a 40 x 40 mil dice as bonded in a standard TO-5 package.

The effect of a metallization on device parameters is studied by metallizing an MOS "P" type structure. In this device, the flat band voltage ( $V_{fb}$ ) is directly related ( $V_{fb} = k + V_t$ ) to the measurable threshold voltage  $V_t$ , or the bias voltage necessary for a 10-microampere current. This value shows a strong dependence on the method of deposition used, which is believed to be a measure of the charge buildup and depletion under various thermal conditions. For the device used, a normal good value is  $V_t = 3$  volts. Using aluminum deposited under various deposition conditions will produce values from 13.4 to 3.4 volts.

Another sensitive vehicle for evaluating electrical effects of a metallization system is transistor units of the matched pair variety. This is because a  $V_{BE}$  match of better than 2 percent over a +125° to -55°C temperature range is specified. In addition, the low current  $h_{FE}$  must be uniformly matched within better than 10 percent throughout processing steps and subsequent aging.

## 8.1 CHROMIUM-GOLD FILMS DEPOSITED BY HIGH VACUUM METHODS

### 8.1.1 Deposition and Control of Evaporated Films of Chromium-Gold

Two deposition methods are being studied: thermal evaporation and low energy inert gas sputtering. In each method it is possible to blend the two metals to form a continuous interface between each metal, if so desired. The interface caused by the blending of the two metals often presents a serious problem in subsequent etching steps, and is used only where necessary for good interlayer adherence. The evaporation procedure for chromium-gold deposition was as follows: The substrate was heated to 250°C before the deposition of the chromium from a basket filament containing a chromium charge. The gold evaporation followed immediately with no appreciable cooling between the two film depositions. The pressure is maintained less than  $10^{-5}$  torr, and the samples are cooled to less than 100°C before exposure to air.

In a series of evaporated films, the thickness of the chromium layer was varied from less than 100 to 3000 Å. The gold thickness varied from 2400 to 6300 Å. The resistivities obtained are shown in Table IV.

The average resistivity is about 6.1 microhm-cm over the range of the data. It is difficult to accurately measure the thickness by optical interference techniques, and thickness variations

TABLE IV  
RESISTIVITY OF EVAPORATED CHROMIUM-GOLD METALLIZATIONS

Thickness. Chromium Å	Thickness Gold Å	Total Å	Resistivity (microhm-cm)
<100	4292	4292	3.6
<100	6300	6300	2.7
350	3770	4120	4.7
625	4850	5475	5.9
665	3535	4200	3.7
665	5400	6065	5.1
700	4330	5030	7.2
860	4330	5290	5.4
900	5040	5940	6.3
1900	5160	7060	10.2
2700	3500	6200	10.4
3000	3650	6650	13.2

across the substrate holder of 25 percent are seen in the gold deposition. However, the data suggests an increased resistivity with the chromium thicknesses greater than 1000 Å. At chromium thicknesses of  $\leq 500$  Å, alloying of the gold with silicon is generally seen early in subsequent processing. Figure 2 shows such alloying on a transistor pattern that occurred during alloying of the gold backing on a wafer.

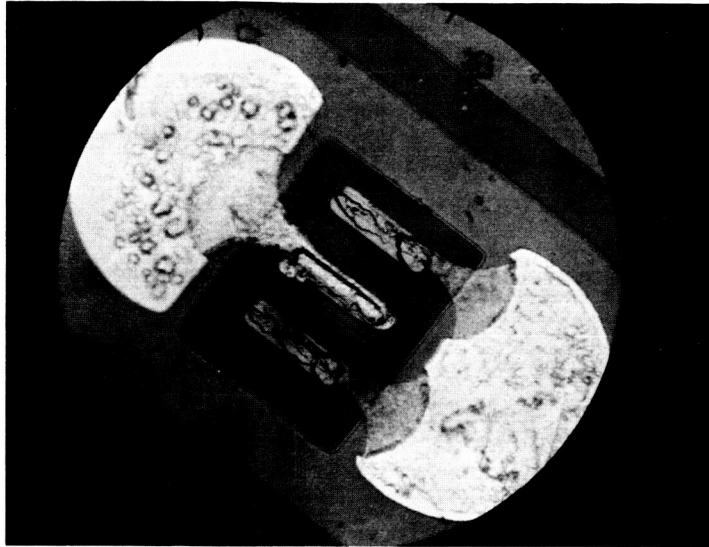


Figure 2. Alloying of Chromium-Gold on a Transistor After Gold Backing Operation ( $T \sim 500^{\circ}\text{C}$ ) Chromium 350 Å Thick

With chromium thicknesses between 500 and 1000 Å, alloying often occurs, although some devices have been made free of this effect. In the most recent tests, chromium thicknesses of  $\geq 1000$  Å have been used, and some of these samples have been completely processed with no alloying observed. However, these units have not been subjected to extensive life tests.

When samples of fine line patterns of metallization on silicon oxide were baked in air at 450°C, changes in the resistivity were seen. There was an initial rise in resistivity that appeared to be related to the chromium thickness, but on overnight baking the resistivity fell to low values. The results of these tests are shown in Table V. The decrease in resistivity observed on these evaporated films is consistent with that found with sputtered chromium-gold where the chromium layer was less than 1000 Å. Apparently the resistivity decrease brought about by temperature annealing of the gold was not offset by an increase that can be caused by the chromium diffusion into the gold. Although the final resistivities of these films suggest that the chromium would provide good metallization, they cannot be used because of subsequent alloying of the gold into the underlying silicon during the alloying of the gold backing.

TABLE V

EVAPORATED CHROMIUM-GOLD METALLIZATION  
(1 mil x 54 mil line tests) Resistivity in microhm-cm vs time of air bake at 450°C

Cr Thickness Å	Total Thickness Å	Resistivity at Time (hrs)				
		t = 0	t = ½	t = 1	t = 1½	t = 18
100	4200	4.1	4.0	5.8	4.3	3.4
665	4200	4.0	9.0	11.4	9.8	2.6
900	5942	8.4	12.0	12.0	11.4	3.4

### 8.1.2 Patterning of Chromium-Gold

The test pattern consists of a group of fine lines  $\frac{1}{2}$  to 2 mils wide and 54 mils long with bonding pads. The successful etching of the  $\frac{1}{2}$ -mil line is dependent upon reducing the undercutting of the resist at any layer to much less than 0.1 mil. In some cases which present serious etching problems, it is possible to use a reverse metal mask of aluminum. With the evaporated films, no troublesome interface film mixing has been used, and a two-step etch of Technistrip (a commercial alkaline cyanide mixture) for the gold, and an "activated" HCl solution for the chromium has been most successful. The "activated" HCl is made up of 1 part HCl and 2 parts water, heated to 80°C. On cooling to 70°C a chromium pellet is added. This is a rapid chromium etch, and usually a quick dip is adequate. Undercutting of the gold is a problem on the films thicker than 1500 Å. An alternate chromium etch is a ceric sulphate-nitric acid mixture at 50-70°C. Either chromium etch is satisfactory for the "normal" evaporated chromium-gold metallizations, but they have been only partially successful for sputtered films. Where aluminum reverse masking was used, any alkaline aluminum etch was successful in lifting the aluminum mask, although this was often a slow process, and in many cases, resulted in the loss of the finer lines because of poor edge definition.

### 8.1.3 Device Characteristics Using Chromium-Gold Metallization

Chromium-gold metallizations using the standard vacuum deposition procedure have given films with good adherence, and satisfactory resistivities for device fabrication. The ohmic contact patterns gave values about 1 ohm between metallized pads onto exposed low resistivity silicon. This compared favorably with aluminum metallization. Improved measurements of these low resistances are necessary before any significance can be attached to the exact

experimental results. The total resistance of the wire bond test vehicle was initially 0.50 ohm. Satisfactory MOS devices were made with  $V_t$  of about 3 volts. These were preliminary tests, which showed only that the  $V_t$  was as good as for the aluminum metallizations.

#### 8.1.4 Reliability Under Stress Aging

The wire bond test vehicles showed an increase from 0.50 to 0.75 ohm over the first 1000 hours in dry air at 300°C. The tests were continued to 1800 hours with no further increase.

### 8.2 CHROMIUM-GOLD SPUTTERING DEPOSITION PROCESS AND EXPERIMENTS

#### 8.2.1 Deposition Procedure and Film Evaluation

Based on the previous experience with sputtered metal films, an arbitrary sputtering process was defined as follows:

- (a) Pump system to less than  $8 \times 10^6$  torr for  $\frac{1}{2}$  hour.
- (b) Heat the substrate to 250°C while Cr and Au are presputtered.
- (c) Presputter Cr and Au for 20 minutes (600 volts) wafers at ground.
- (d) Open shutter and sputter Cr (600 volts) for 8 minutes (after 3 minute turn on -50 volts bias, heater off and cooling air on.
- (e) In last 3 minutes of Cr time turn on Au (-700 volts) and cosputter 3 minutes. (3 minute interface.)
- (f) Cr off and substrates ground, substrates cold, and sputter Au for 60 minutes.

Variation of this procedure included:

Cr deposition time - 2, 3, 5, 7, 9 and 12 minutes, and  
Cr-Au interface time - 0, 1, 3 and 7 minutes.

The chromium deposition rates were 175 to 250 Å/minute  
and the Au rate was approximately 125 Å/minute.

Seventeen deposition runs were made in which the Cr thickness and Cr-Au interface type and thickness were varied in a systematic manner. Each run contained 10 silicon wafers which were heated to 250°C during a portion of the sputtering process as outlined in the sputtering procedure. Several wafer experiments were designed into each run.

Positions 1 and 3: Thermally oxidized wafers for ohms per square and later use.

Position 2: Thermally oxidized wafer with masked edge for total thickness.

Positions 4 and 6: Thermally oxidized wafers for etching and measuring  $\Delta R$  with 450°C air bakes.

Positions 5 and 7: Thermally oxidized wafers for etching and measuring Cr thickness.

Position 8: Wafer with no oxide for alloy test.

Positions 9 and 10: Glass substrates for visual tests on Au diffusion.

Due to thickness nonuniformity over the complete 4 x 5 inch substrate holder, it was necessary to determine a range of Cr thickness for each run and in some cases the Cr thickness overlapped from one run to the next. The thickness range on wafers 5 and 7 (above) has been found to be representative of all wafers in a given run.



## 8.2.2 Results and Discussion

### 8.2.2.1 Resistance of Sputtered Chromium-Gold Films

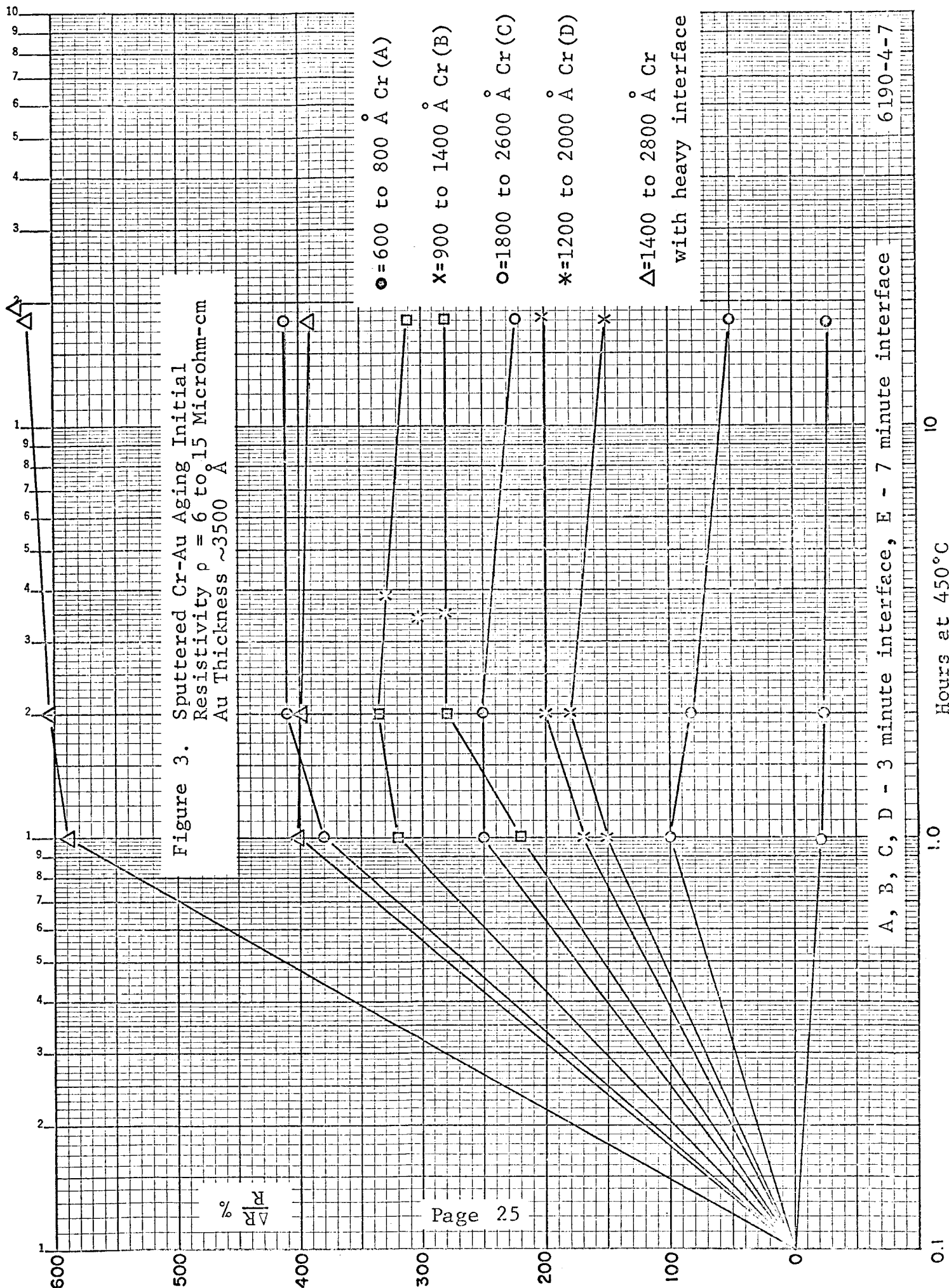
The as-deposited resistivity of the sputtered Cr-Au composite films ranged from 6 to 15 microhm-cm. The variation in resistivity was assumed to be related to the relative amounts of Cr and Au in the composite film but this point was not checked as, the ohms per square, the Cr thickness, and the total thickness were not measured on the same wafer.

Measurements made of the resistance of the etched patterns after various times in air at 450°C showed a large increase in resistance. The results are shown in Figure 3. The thicknesses shown are those taken from wafers from positions 5 and 7 of each run. Note that the resistance of the films with chromium layers thicker than approximately 1000 Å increase 200 to 600 percent within 1 hour at 450°C, and then increase less rapidly thereafter. The films with heaviest interface tend to increase more. A light or no interface reduced the rate of reaction as indicated by the resistance changes. Unfortunately the adherence of the Au to the Cr then becomes marginal.

Schnable and Keen<sup>(1)</sup> have studied Cr-Au composite films and found resistance increases were as large as 170 percent after 50 hours at 350°C in nitrogen ambients. The results found on these sputtered Cr-Au films are similar and the magnitude of the resistance increase is too large to be a simple oxidation of the Cr from under the Au. The more plausible explanation lies in solid state reactions and diffusion of the Au into the Cr (or Cr into the Au) which results in the formation of a high resistance intermetallic compound.

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<sup>(1)</sup> G. L. Schnable and R. S. Keen, "Study of Contact Failures in Semiconductor Devices," RADG-TR-66-165, Final Report, April 1966.



#### 8.2.2.2 Gold-Silicon Reaction

In active device fabrication, the metallization must make ohmic contact to the Si at the emitter, base and collector contacts. If one of the metals in the system is gold, it cannot be permitted to come into contact with the Si at temperatures near the Au-Si eutectic. If this happens, the transistor may be degraded by (a) Au doping reducing carrier lifetime or (b) gold alloying down into a junction region, degrading it severely. To test for this effect, a Si wafer with the oxide removed was sputtered with metal in each run. Visual indications of the Au-Si alloying were sought after various times at 450°C. A glass wafer was also included in each run so that the base or substrate interface of the deposited film could be observed for changes. The expected behavior here, of course, was that the time required for the alloying even to occur would increase with Cr thickness. This was not observed; all the "no oxide" wafers alloyed within  $\frac{1}{2}$  hour at 450°C even though the thickest Cr film was 1500 to 1900 Å.

However, the glass substrate films did exhibit the anticipated behavior. That is, the time required at 450°C for the Au color to show on the back of the Cr-Au deposit increased with Cr thickness. The combined results indicates either that pinholes in the Cr film speed up the Au-Si reaction or that sufficient gold diffuses and starts the reactions before a visible change occurs. It is possible that thicker pinhole free Cr films would inhibit the Au-Si alloy from forming but this trade-off would result in higher resistance interconnects and increased difficulty in etching fine lines.

#### 8.2.2.3 Chromium-Gold Adherence

Regardless of the thickness, the sputtered Cr films adhere well to the SiO<sub>2</sub> but the adherence of the Au to the Cr depended on the amount of interface between the two. The adherence (as indicated

by a probing test) of the Au before baking is sufficient, but certain films start to peel in 450°C stress tests. In particular, the Au on the zero and 1 minute interface films started to lift off the Cr within 1 hour at 450°C.

#### 8.2.2.4 Chromium-Gold Patterning

A Cr pellet activated HCl solution was used to etch the films. This etch was discussed in Section 8.1.1 and an exact etching procedure was followed precisely to obtain repeatable results.

The following observations were made on the seventeen depositions:

- (a) Etchability of the Cr-Au decreases as the Cr thickness increases until Cr thicker than approximately 2500 Å could not be etched in a repeatable manner, particularly if the substrates are heated to 250°C during deposition.
- (b) Thickness of Cr was important in etching; however, the substrate temperature during deposition affected the etching also. For example, one run with Cr thickness = 1600 to 1900 Å was inadvertently heated at 250°C for some 15 minutes longer than the standard time used and this wafer could not be etched. The Cr and Au seemed to be alloyed in large spots and neither Technistrip, aqua regia, or activated HCl would etch these areas.
- (c) The films with heavy interface did not etch as easily as those without but the effect was second order as compared to Cr thickness and substrate temperature.

The most common etch problem is described by undercutting of the Au by a lateral etch of the Cr. This occurs in the etching of the Cr while attempting to etch clean approximately the last third of the wafer. In some cases, areas of Au could not be removed in Technistrip or aqua regia and the appearance of these areas seemed to be related to excessive temperature during deposition.

### 8.3 HIGH CURRENT DENSITY TESTS WITH CHROMIUM-GOLD METALLIZATIONS

High current density tests of the chromium-gold metallization at 175°C ambient have been extended to a maximum total life of 2500 hours per sample. The results are tabulated in Table VI (using a 50 percent confidence level)\*.

TABLE VI  
MEAN TIME TO FAILURE FOR CHROMIUM-GOLD METALLIZATION  
CURRENT DENSITY AND RESISTIVITY

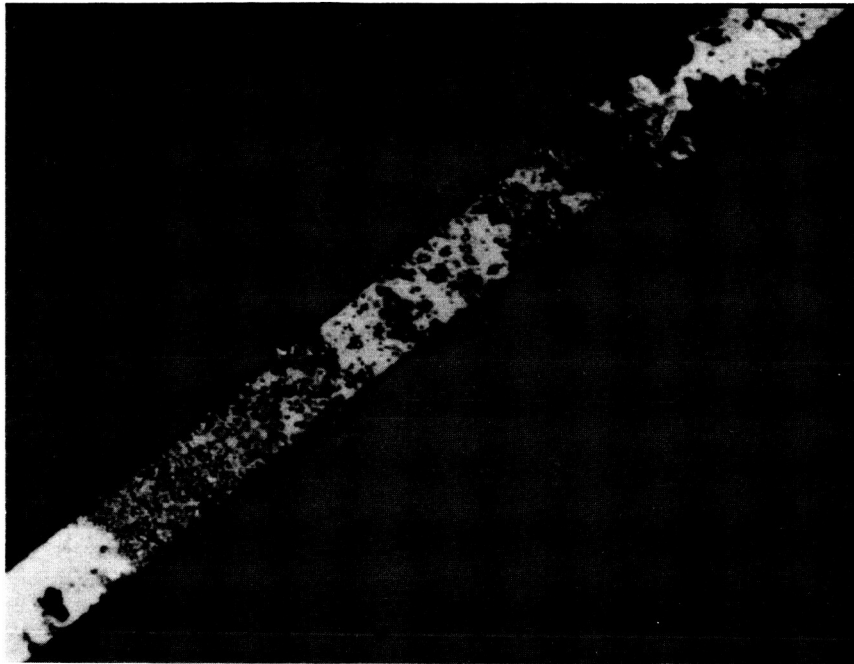
J =	$10^6 \text{ A/cm}^2$	$2 \times 10^6 \text{ A/cm}^2$	$3.2 \times 10^6 \text{ A/cm}^2$
$\rho = 4.3_{\mu} \Omega\text{-cm}$ 665 Å Cr	30,000 hrs.	4,400 hrs.	----
$\rho = 8.1_{\mu} \Omega\text{-cm}$ 2800 Å Cr	30,000 hrs.	650 hrs.	----
$\rho = 7.2_{\mu} \Omega\text{-cm}$ 700 Å Cr	----	1,000 hrs.	2 hrs.

\*The previous reports have given data calculated using a 95 percent confidence level. The change to 50 percent was made to include additional data from experiments where the time to failure for half the sample was determined.

The failure mechanism for the chromium-gold metallization is seen in the examination of the failed units. The failures were predominantly due to an increased resistance of the metal strip, caused by an alloying or diffusion of the chromium and gold layers. This high resistance mode has been discussed in Section 8.1 and 8.2 in reference to thermal treatments (450°C and no current) of these metallizations. The resistance increase is such that the strip resistance doubles in about 500 hours with the higher resistivity metallizations. However, these strips will still carry current and are acting as resistors at 2500 hours. A typical case is shown in Figure 4.

This failure mode appears randomly along the strip on the samples which have an "average calculated film temperature" of 220° to 285°C. It has not yet appeared in the longer life samples which are operating at about 185°C. The two failures (out of 30 samples) at the lower temperature are typical burn-out opens, as shown in Figure 5.

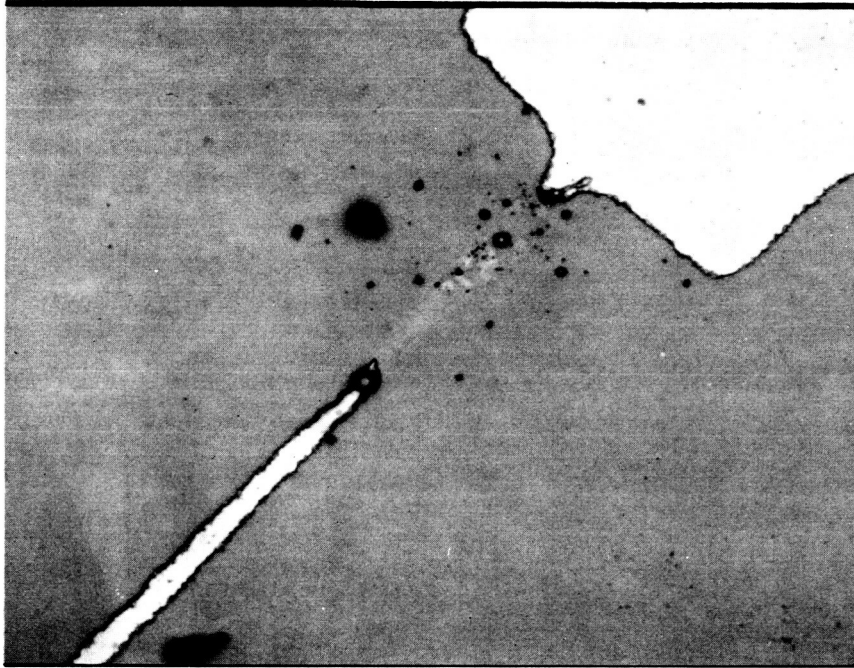
Each group was tested at 1 and  $2 \times 10^6$  A/cm<sup>2</sup>. The current through each unit is monitored by daily measurements of the voltage drop across the individual external resistor in series with the sample. By observing this daily measurement, changes in the sample resistance can be seen, as well as failures due to open connections or failure of the interconnect strip. In the case of the chromium-gold samples, there have been no failures due to opening of the strip. However, there has been an increase in the strip resistance in the case of the  $2 \times 10^6$  A/cm<sup>2</sup> test. This increase was verified by removal of the samples and measurements made with the sensitive bridge. For example, when the current measurement indicated an average increase of 14 ohms, the bridge indicated an average of 13 ohms.



800X

Figure 4. Photomicrograph of Cr Au  $2 \times 10^6$  A/cm<sup>2</sup>  
(Resistance increased from 12 to 50 ohms  
after 764 Hours)

6317-6-7



800X

Figure 5. Photomicrograph of Cr Au Showing  
Typical Burn-out Opens

6318-6-7



The sample of 10 at  $1.9 \times 10^6$  A/cm<sup>2</sup> showed an average increase at 100 hours of 3.3 ohms or 25 percent, with some individuals showing nearly a 100 percent increase. The estimated average film temperature (oven + I<sup>2</sup>R drop) was 224°C. At 250 hours, 50 percent of the sample had increased more than 100 percent, with an average increase of 5.5 ohms or 44 percent. A constant voltage source is used; so the current fell slightly to  $1.84 \times 10^6$  A/cm<sup>2</sup>, although the estimated average film temperature rose to 239°C. At 500 hours, all the samples had increased greater than 100 percent, with an average increase of 31 ohms or 250 percent. The current level had fallen to  $1.67 \times 10^6$  A/cm<sup>2</sup>, with an estimated average film temperature of 259°C. Despite the increasing film temperature, there were no failures from open or fused interconnection strips. Samples of this metallization (2800 Å chromium) in test at  $1 \times 10^6$  A/cm<sup>2</sup> showed a possible increase of approximately 0.6 ohm or 4.5 percent after 500 hours. The film temperature was estimated at 186°C.

Samples with a thinner chromium layer (665 Å) on thermal oxide showed an increase after 500 hours of 0.8 ohm or 5.7 percent at  $2 \times 10^6$  A/cm<sup>2</sup>, and 0.3 ohm or 2.5 percent at  $1 \times 10^6$  A/cm<sup>2</sup>. The film temperatures are estimated at 186° and 180°C, respectively.

During the measurements of the strips using the bridge, the 1-mil lines were also checked. These had no current through them and were, therefore, near the oven temperature of 175°C. These samples showed a possible increase of 0.2 ohm or 3 percent after 500 hours.

If one plots the increase in resistance after 500 hours as a function of the reciprocal of the absolute temperature, as shown in Figure 6, the activation energy or temperature coefficient of the process is determined as about 42,000 cal/mole. This is very near the value (41,700 cal/mole) given as the self diffusion coefficient of gold.<sup>(1)</sup> It is doubtful if the temperatures of the films and the

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(1) Feissel & Armstrong, Transactions of the AIME, Vol. 230, (4), 867 (1964).

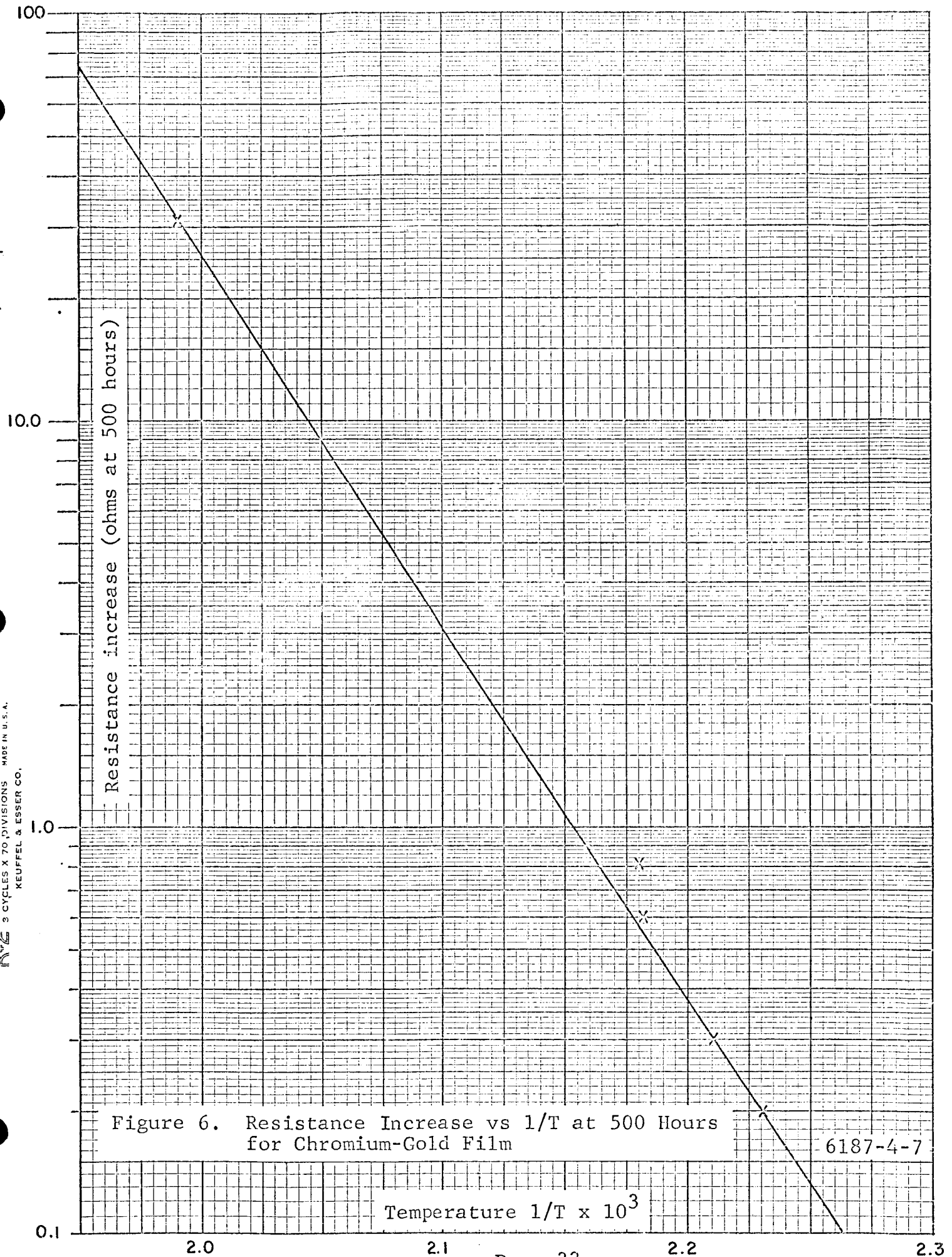


Figure 6. Resistance Increase vs  $1/T$  at 500 Hours for Chromium-Gold Film

6187-4-7

other parameters are known well enough to cite these experiments as proof of any specific theory, but the results are certainly suggestive that the resistance increase is a diffusion limited process.

#### 8.4 CHROMIUM-GOLD METALLIZATION - CONCLUSIONS

Chromium-gold interconnection metallization has so far shown to be incompatible in transistor and integrated circuit fabrication because of the following results.

- (a) Chromium thicknesses greater than 2000 Å are required to prevent gold diffusing through the chromium and alloying with the underlying silicon within a  $\frac{1}{2}$  hour time period at 450°C.
- (b) The resistivity of Cr + Au films with chromium layers thicker than 1000 Å increase more than 200 percent within 1 hour at 450°C.
- (c) Chromium-gold films with a chromium layer thicker than 2000 Å cannot be etched in a repeatable manner.
- (d) Substrate temperature during deposition of greater than 250°C or a significant interface between the chromium and gold layer increases the rate of resistivity change and also increases the difficulty with etching. Such interfacing is required in order that reliable adhesion exists between the chromium and gold for all methods of film deposition and subsequent processing.

The etching problem by itself, while troublesome, would not render the Cr-Au system incompatible. Undoubtedly, further experimentation with different etch solutions and procedures could overcome this problem. Even if this did not prove to be true, a reverse masking process might be used.

The more fundamental problem is in the Cr-Au system itself and the rapid rate of solid state diffusion and reactions indicated by the temperature instability in resistance. The results here show that even 2000 Å of Cr is not sufficient to stop the alloying of Au with Si in short term 450°C bakes. Even if the Cr could be increased in thickness to inhibit the Au diffusion to the Si, the associated large resistance increase could not be tolerated in an operational circuit.

The use of Cr-Au metallization is not recommended for use in a high reliability integrated circuit technology which requires 450°C - ½ hour air bakes in the processing. However, in a transistor-circuit fabrication process which does not require temperature-times as high as 450°C - ½ hour, Cr-Au may be a compatible metallization system. Such a process could be envisioned as a low temperature die bonding process combined with ultrasonic wire bonding (or low temperature wire bonding). The die could then be encapsulated in a low temperature sealing plastic.

## SECTION IX

### 9.0 MOLYBDENUM-GOLD

Molybdenum-gold systems are more stable at elevated temperatures than the chromium-gold system, but the molybdenum is readily dissolved during a boiling water high stress reliability test. It appears that this will not be a basic limitation, especially in extra-layer passivated or multilayer structures where a glass passivation layer can be used to protect the molybdenum. The etching of the molybdenum system is much simpler than the corresponding chromium system. The same test vehicles as previously discussed in the section on chromium-gold were used with the molybdenum-gold experiments.

#### 9.1 DEPOSITION AND CONTROL OF EVAPORATED FILMS OF MOLYBDENUM-GOLD

A "standardized" vacuum deposition cycle has been developed for molybdenum-gold. The substrate is heated to 260°C and about 1000 Å of molybdenum is deposited using electron beam heating of a molybdenum target, followed by about 300 Å of gold (substrate temperatures as high as 320°C produced no significant change in resistivity). The substrate is cooled to less than 100°C, and about 3000 Å of additional gold is deposited. The pressure is maintained less than  $10^{-5}$  torr, and the samples are cooled to less than 40°C before the system is opened. The interface, formed by blending the two metals, can be resistant to the normal etchants used, and cause difficulty in directly etching fine patterns.

The thickness of the molybdenum was varied from <100 to 2080 Å, as shown in Table VII.

TABLE VII  
EVAPORATED MOLYBDENUM-GOLD METALLIZATIONS  
RESISTIVITY IN MICROHM-CM VS MOLYBDENUM THICKNESS

Mo Thickness	Total Thickness Mo & Au	Resistivity as Deposited
<100 Å	2800 Å	2.7
360	2661	2.9
330	6880	3.9
390	2668	4.0
750	3000	4.1
840	2800	4.3 (320°C substrate)
1200	3810	4.1 (320°C substrate)
1400	4100	4.1
2080	3380	4.9

## 9.2 DEPOSITION AND CONTROL OF SPUTTERED MOLYBDENUM-GOLD FILMS

A series of molybdenum-gold films was sputtered onto oxidized silicon wafers. A standard triode sputtering procedure has been established as follows:

- (1) Let system pump to less than  $8 \times 10^{-6}$  torr for  $\frac{1}{2}$  hour.
- (2) Heat substrate to 250° to 300°C and hold for  $\frac{1}{2}$  hour.  
The time may be part of the pumpdown.
- (3) Backfill to 1 micron with argon and hold pressure stable.
- (4) Presputter with substrates protected by shutter.

- (5) Open shutter and sputter Mo with 600 V for 6 minutes. After 3 minutes the bias is set at -50 volts and the substrate cooling air turned on. The observed rate has been 150 to 170 Å/minute.
- (6) If an interface is desired, cosputter with 1000 volts on the gold target and 600 volts on the molybdenum target, with -50 volt bias on substrate. The deposition rate is typically 200 to 250 Å/minute.
- (7) Turn off bias, ground substrate, force cool substrate holder. Sputter gold at 600 volts for 60 minutes.

The sputtered molybdenum films gave resistivities of approximately 15 microhm-cm. Molybdenum-gold films varied from 8 to 12 microhm-cm. Any correlation between resistivity and deposition parameters was masked by the variability of the deposited film found across the substrate holder. Thickness variations across the 3 x 4 inch holder as high as 30 percent have been observed. A typical result is  $4500 \text{ Å} \pm 1200 \text{ Å}$ , or 27 percent. This variation was dependent on location of the wafer in the holder and independent of the metal being sputtered.

### 9.3 PATTERNING OF MOLYBDENUM-GOLD

The test pattern was the same as that used in the chromium-gold experiments discussed previously. This pattern was etched from the deposited films after appropriate photoresist masking as follows: The gold is removed by Technistrip and a warm nitric-acetic-phosphoric acid mixture was used for the molybdenum. In the case where an intermetallic interface compound was prepared by codeposition, the interface was rapidly removed by the molybdenum etch at 40° to 45°C. If the etch was used at room temperature, the reaction at the interface

was very slow, and often a surface film was left at the Mo-SiO<sub>2</sub> interface. The definition was excellent down to the ½-mil line widths.

#### 9.4 DEVICE CHARACTERISTICS USING MOLYBDENUM-GOLD METALLIZATION

Molybdenum-gold metallizations using the standard vacuum deposition procedure have given films with satisfactory adherence and resistivities. Wire bonding to the gold layer has been satisfactory with films containing at least 1000 Å of gold. The ohmic contact vehicles gave normal value of about 1 ohm between two adjoining silicon contacts, comparing favorably with aluminum. The wire bond measurement was initially 0.54 ohm. Satisfactory MOS devices were made with the  $V_t$  measured as 4.3 volts for the evaporated film and 3.4 volts for the sputtered film.

To observe any effects of the sputtering process and the molybdenum-gold metallization on transistor quality, two NPN transistor wafers with  $X_{jB} \sim 2$  microns,  $R_s = 160$  ohms per square ( $\rho_p \sim 0.03$  ohm-cm) were metallized using the standard sputtered Mo-Au. The various transistor parameters are summarized in Tables VIII and IX.

From the experiments we note the following:

- (a) A slight drop in  $h_{FE}$  may be caused by the sputtering, but the sputtering process used did not severely degrade the transistors.
- (b) For low resistivity "P" base ( $\rho_p \sim 0.03$  ohm-cm) Mo-Si ohmic contacts may not be a problem if the Si is clean. Comparing the  $V_{BES}$  values with Al contacted wafers from the same diffusion lot, the Mo-Au is only slightly



TABLE VIII  
SPUTTERED MOLYBDENUM-GOLD  
3/14/67 RUN

	$h_{FE}$ at $V_c = 1$ V 0.1 mA                  1 mA		$I_c = 10$ mA $I_b = 1$ mA $V_{BES}$
Before Metal- lization	30	40	
After Sputtering Mo-Au*	27	28	0.9 V
After Cleaning	28	30	0.9 V

\* $h_{FE}$  extremely uniform over whole wafer.

TABLE IX  
SPUTTERED Mo-Au AFTER EVAPORATED ALUMINUM  
ON TRANSISTORS

	$h_{FE}$ at $V_c = 1$ V 0.1 mA                  1 mA		$I_c = 10$ mA $I_b = 1$ mA $V_{BES}$
Before Metal- lization	40	45	
After Sputtered Mo-Au	30	35	1.05 V
After Cleaning	35	40	1.1 V

higher. The lack of agreement in  $V_{BES}$  (Tables VIII and IX) on the two wafers is due to poor Mo-Si contact. The area of Mo-Si contacts will continue to be investigated.

- (c) No indication of Au-Si alloying was seen after 450°C for 15 minutes.

The excellent thermal properties of the molybdenum-gold system, its compatibility to fine line patterns by direct etching, and its demonstrated high current performance suggest that the system be studied in greater detail. Specifically, the high resistivity problem of the sputtered material should be investigated, and high current tests at lower ambient temperatures (~100°C) should be considered. The combination of the favorable properties suggest the extension of the molybdenum-gold metallization to multilayer test vehicles. Also, high temperature tests should be considered to elucidate the mechanism of the failures seen, and establish some temperature limits for the system.

## 9.5 RELIABILITY UNDER STRESS AGING

Wire bond test vehicles of evaporated molybdenum-gold films showed a slight decrease in resistance from 0.54 to 0.46 ohm in 1000 hours at 300°C in dry air. Heating in air at 450°C for as long as 18 hours showed a similar slight decrease in resistivity with the evaporated films. The recent sputtered films were also heated in air at 450°C. Again, a slight decrease (approximately 0 to 25 percent) in the resistance occurred in the first 2 hours. On overnight baking at 450°C in air, the resistance increased slightly to values slightly higher (0 to 25 percent) than initial. The exception of this was the case where an estimated 1200 to 1500 Å of interface was blended into the deposition. This film showed an initial decrease (approximately 25 percent) in resistance, but after

overnight at 450°C in air, the 0.5- and 1-mil lines were completely open. Visual examination suggests a reaction of the molybdenum and gold or oxidation of the molybdenum or both. The finer lines were completely removed, with an outline on the oxide surface where the metal had originally been. The 2-mil lines were reduced to perhaps 0.5 mil by the reaction. In the case where the Mo was  $\leq 800$  Å thick, the 18-hour bake caused the formation of pinholes in the film, the result of localized reaction with the substrate.

#### 9.6 HIGH CURRENT DENSITY TESTS WITH MOLYBDENUM-GOLD METALLIZATION

The high current density tests at 175°C ambient have been continued to a maximum life per sample of 2500 hours. Sputtered molybdenum-gold metallizations were added later to the test and a stress level of about  $3.75 \times 10^6$  A/cm<sup>2</sup> was included. The results are shown in Table X (using a 50 percent confidence level).

TABLE X  
MEAN TIME TO FAILURE OF MOLYBDENUM-GOLD METALLIZATION  
AS A FUNCTION OF CURRENT DENSITY AND RESISTIVITY  
AMBIENT = 175°C

J =		10 <sup>6</sup> A/cm <sup>2</sup>	2x10 <sup>6</sup> A/cm <sup>2</sup>	3.7x10 <sup>6</sup> A/cm <sup>2</sup>
ρ (Microhm-cm)				
Evap- orated Films	3.3	30,000 hrs.	30,000 hrs.	----
	4.1	30,000 hrs.	13,000 hrs.	----
	4.3	11,200 hrs.	11,200 hrs.	----
	5.1	30,000 hrs.	20,000-30,000	420 hrs.
Sputter- ed Film	7.2	----	7,900 hrs.	71 hrs.
	9.4	----	310 hrs.	----
	10.4	----	10 hrs.	----

It is apparent that the higher resistivities are associated with the reduced lifetimes. The sputtered films, with their higher resistivities, are poorer than the evaporated films. However, if they are compared at equivalent "average calculated film temperatures," they appear equal. The  $3.75 \times 10^6$  A/cm<sup>2</sup> and  $\rho = 5.1$  microhm-cm gave 241°C and 420 hours; while the  $1.75 \times 10^6$  A/cm<sup>2</sup> and  $\rho = 9.4$  microhm-cm gave 243°C and 310 hours.

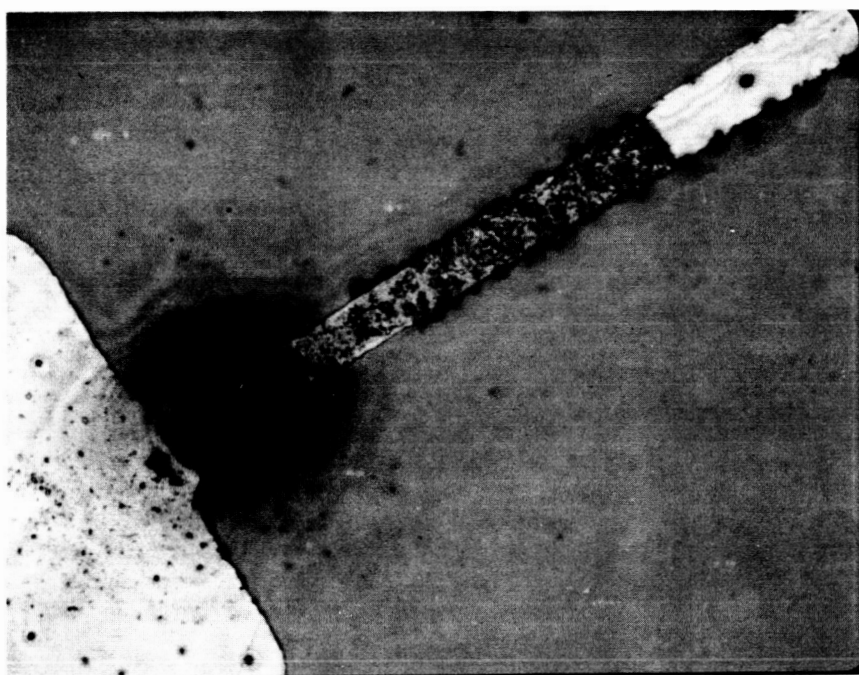
The failure mechanism appears complicated in the molybdenum-gold system. The earlier failures, and particularly those in the evaporated metal system, appear predominantly as opens. In some cases, a high resistance mode is observed prior to failure. This is illustrated by the example in Figure 7.

This failure occurred at the positive end of the strip, and showed the moly remaining under scattered discontinuous gold particles. A more obvious case of gold fusing is shown in Figure 8.

This occurred in 175 hours with a current density of  $1 \times 10^6$  A/cm<sup>2</sup> and an "estimated average film temperature" of 180°C. This is the single failure in a group of nine samples with an accumulated life of 19,064 hours (mtf = 11,200 hours).

The sputtered molybdenum-gold films have perhaps 300 Å of interface blended during the deposition. In addition, the target arrangement allows the cross contamination of the two metal sputtering targets. These films are characterized by a high resistivity and show predominantly a high resistance type of failure of the type shown in Figure 9.

This film showed a resistance of 39.8 ohms (initial = 8 ohms) after 480 hours at  $3.65 \times 10^6$  A/cm<sup>2</sup> and 175°C ambient. The failure occurred at the positive end of the strip and shows "alloying" of



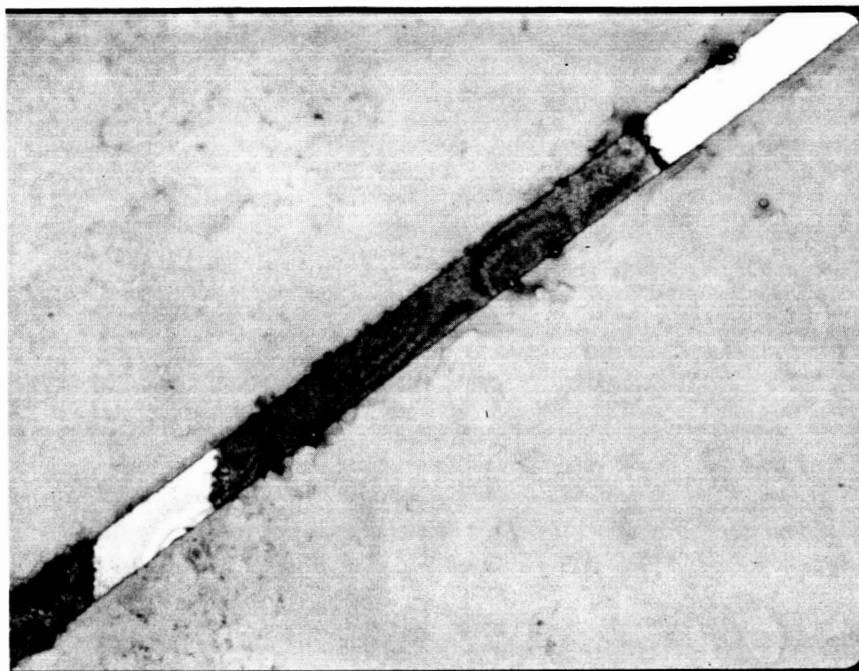
800X

Figure 7. Evaporated Mo-Au  $3.75 \times 10^6 \text{ A/cm}^2$   
480 hours 102 ohms  $t \sim 241^\circ\text{C}$  Resistance



680X

Figure 8. Evaporated Mo-Au  $10^6$  A/cm<sup>2</sup>  
175 hours



800X

Figure 9. Sputtered Mo-Au  $3.65 \times 10^6$  A/cm<sup>2</sup>  
480 hours

6321-6-7

the metals. In the sputtered (blended) films, this is seen at  $2 \times 10^6$  A/cm<sup>2</sup> and a film temperature of 202°C, while in the evaporated films it appeared only at ~240°C.

This behavior was also seen in the thermal-only stressing, where the blended sputtered films showed an increased resistivity on baking in air at 450°C, and after overnight were completely open.

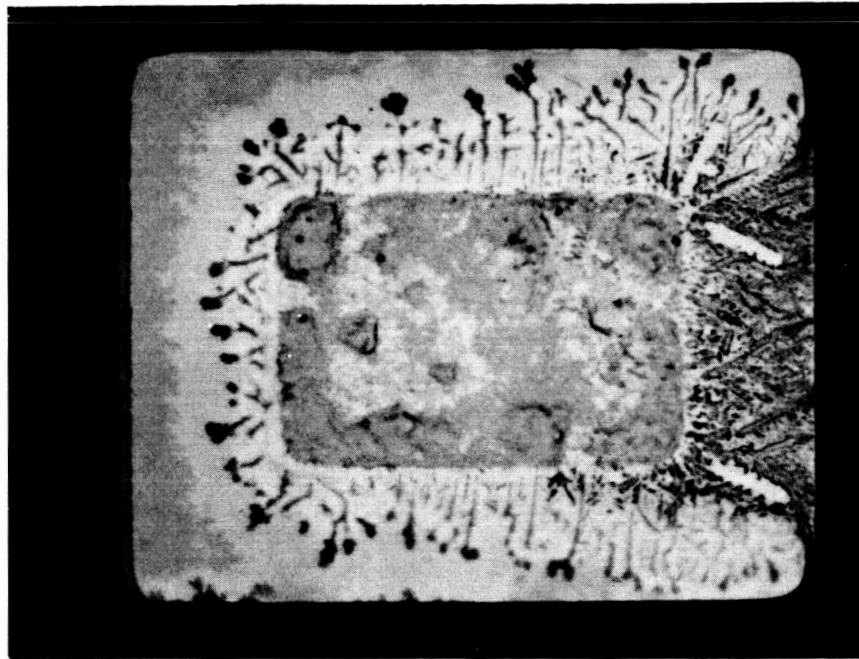
Thus it appears that there is a mode of failure involving alloying or reaction of the molybdenum and gold layers, which, although it appears only at the higher temperatures, higher stresses, and longer times, may be significant.

An additional possible weakness may also develop in the alloying of the gold with the silicon by penetration of the molybdenum layer. In cases where the molybdenum is less than 2000 Å thick, reaction of the gold with the silicon has been observed after overnight at 450°C. This is shown in Figure 10, which shows a 4 x 4 mil moly-gold contact deposited over a 2 x 2 mil opening etched through 10,000 Å of thermal oxide to the silicon substrate. In this case, the 1400 Å of molybdenum-alloyed extensively after overnight at 450°C, although for several hours no significant reaction was observed. Tests with ~2000 Å of molybdenum have shown greater resistance to alloying, and no difficulties have been encountered in normal 450°C processing or in subsequent short term 300°C tests.

#### 9.7 MOLYBDENUM-GOLD METALLIZATION CONCLUSIONS

The molybdenum-gold metallization system has not as yet progressed through the many tests used in evaluating chromium-gold so concrete conclusions cannot be formulated at this time. The following statements concerning moly-gold can be made:





640X

Figure 10. 1400 Å Mo + 1960 Å (Failed after 48 hours  
at 450°C)

6322-6-7

- (a) Molybdenum-gold is considerably more stable at elevated temperatures than the chromium-gold.
- (b) Molybdenum-gold, although not without problems, is considerably easier to etch into fine line interconnections than chromium-gold.
- (c) Using a minimum of interfacial mixing, the moly-gold films do not undergo the large increases in resistivity observed with Cr + Au due to high temperature baking.
- (d) Thick interfacial mixing results in severe film degradation if the film is subjected to high temperature air bakes.
- (e) No alloying of gold into the underlying silicon will occur if the molybdenum film is free of pinholes over the exposed silicon.
- (f) Mean time to failure as predicted from the high current density studies show that molybdenum-gold is superior to any other metallization system evaluated to date.